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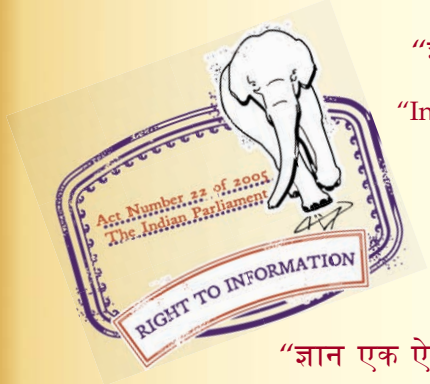

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Indian Standard

SPECIFICATION FOR BENZOYL CHLORIDE, TECHNICAL

UDC 547·582·1

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 1

July 1972

Indian Standard

SPECIFICATION FOR BENZOYL CHLORIDE, TECHNICAL

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SPECIFICATION FOR BENZOYL CHLORIDE, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 5 November 1971, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 Benzoyl chloride is used as an intermediate for introduction of benzoyl group in organic compounds. It is also used in the manufacture of dye intermediates and benzoyl peroxide.

0.3 Benzoyl chloride vapours are lachrymatory and hence sufficient care should be taken in handling of this material.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for benzoyl chloride, technical.

2. GRADES

2.1 The material shall be of the following grades:

Grade 1 — intended for use in the manufacture of organic peroxides, and

Grade 2 — intended for use in dye-stuff and drug industries and for other industrial purposes.

3. REQUIREMENTS

3.1 Description — The material shall be colourless to yellow liquid free from suspended matter. It shall fume in moist air and shall be soluble in

*Rules for rounding off numerical values (*revised*).

most organic solvents. It shall be slowly but almost completely soluble in sodium hydroxide solution.

3.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of the Appendix is given in col 5 of the table.

TABLE 1 REQUIREMENTS FOR BENZOYL CHLORIDE, TECHNICAL

Sl No.	Characteristic	Requirement		Method of Test (Ref to Cl No. in Appendix A)
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Relative density* at 27°C/27°C	1·206 to 1·212†	1·204 to 1·214†	A-2
ii)	Benzoyl chloride content, percent by mass, *Min*	99·0	95	A-3
iii)	Free hydrochloric acid, percent by mass, *Max*	0·20	0·5	A-3.4
iv)	Free benzoic acid, percent by mass, *Max*	0·25	—	A-3.5
v)	Residue on evaporation, percent by mass, *Max*	0·05	0·1	A-4

*Relative density is the term adopted for specific gravity with water as reference substance by the International Organization for Standardization (ISO).

†The relative density determined at any temperature within the range of 25 to 35°C can be adjusted to 27°C/27°C by using a correction factor of + 0·000 7 for every degree Celsius fall and — 0·000 7 for every degree Celsius rise in temperature.

4. PRECAUTIONS IN HANDLING AND STORING

4.1 Owing to the hazardous nature of the material, care shall be taken at all stages of its handling and storage.

5. PACKING AND MARKING

5.1 Packing — The material shall be supplied in well stoppered glass carboys or other suitable containers as agreed to between the purchaser and the supplier.

5.1.1 It shall be stored in a cool and dry place.

5.1.2 The containers for storing and transport shall additionally conform to the latest requirements laid down by the Chief Inspector of Explosives, Government of India.

5.2 Marking

5.2.1 The containers shall be marked with the words 'WARNING! HAZARDOUS LIQUID — LACHRYMATORY' and 'THIS WAY UP' in capital letters.

5.2.2 The containers shall also be marked with the following:

a) Name and grade of the material;
b) Manufacturer's name and his recognized trade-mark, if any;
c) Mass of the material in the container; and
d) Lot or batch number, in code or otherwise.

5.2.3 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. Presence of this mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard, under a well-defined system of inspection, testing and quality control during production. This system, which is devised and supervised by ISI and operated by the producer, has the further safeguard that the products as actually marketed are continuously checked by ISI for conformity to the standard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

6. SAMPLING

6.1 Representative samples of the material shall be drawn and their conformity to the requirements of this standard judged as prescribed in Appendix B.

APPENDIX A

(*Clause* 3.2)

METHODS OF TEST FOR BENZOYL CHLORIDE, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF RELATIVE DENSITY

A-2.0 Outline of the Method — Masses of equal volumes of the material and water at the same temperature are compared using relative density bottle.

*Specification for water, distilled quality (*revised*).

A-2.1 Apparatus

A-2.1.1 *Relative Density Bottle* — 25 ml capacity.

A-2.1.2 *Water-Bath* — maintained at 27·0 ± 0·2°C.

A-2.1.3 *Thermometer* — any convenient thermometer of a suitable range with 0·1 or 0·2 deg subdivisions.

A-2.2 Procedure — Clean and dry the relative density bottle, weigh and then fill with recently boiled and cooled water at 27°C. Fill to overflowing by holding the relative density bottle on its side in such a manner as to prevent entrapment of air bubbles. Insert the stopper and immerse in the water-bath. Keep the entire bulb of the bottle completely covered with water and hold at that temperature for 30 minutes. Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry, cool and weigh. Again clean and dry the relative density bottle. Using the material under test, proceed exactly as in the case of water and weigh the bottle with the material.

A-2.3 Calculation

$$\text{Relative density at } 27°C/27°C = \frac{A - B}{C - B}$$

where

A = mass in g of the relative density bottle filled with the material at 27°C,

B = mass in g of the clean and dry relative density bottle, and

C = mass in g of the relative density bottle filled with water at 27°C.

A-3. DETERMINATION OF BENZOYL CHLORIDE CONTENT

A-3.0 Outline of the Method — A known amount of standard sodium hydroxide solution is added to a known amount of the material, allowed to dissolve, and then the excess of sodium hydroxide solution is titrated with standard sulphuric acid to determine total acidity. Total chlorides is estimated by argentometry. Free chlorides are estimated separately after extracting benzoyl chloride with ether.

A-3.1 Reagents

A-3.1.1 *Standard Sodium Hydroxide Solution* — 1 N.

A-3.1.2 *Standard Sulphuric Acid* — 1 N.

A-3.1.3 *Phenolphthalein Indicator Solution* — 0·5 percent (*m/v*). Dissolve 0·5 g of phenolphthalein in 100 ml of rectified spirit (*see* IS : 323-1959 *) and make it faintly pink by the addition of dilute sodium hydroxide solution.

A-3.1.4 *Silver Nitrate Solution* — approximately 0·1 N.

A-3.1.5 *Standard Ammonium Thiocyanate Solution* — 0·1 N.

A-3.1.6 *Ferric Alum Indicator Solution* — saturated.

A-3.1.7 *Nitrobenzene*

A-3.1.8 *Nitric Acid* — sp gr 1·40 (*see* IS : 264-1968†).

A-3.1.9 *Solvent Ether* — *See* IS : 336-1964‡.

A-3.2 Procedure

A-3.2.1 Weigh accurately about 2 g of the material in a glass stoppered flask, add 50 ml of standard sodium hydroxide solution, stopper and allow to stand with frequent agitation until the material is dissolved. Titrate the excess of sodium hydroxide with standard sulphuric acid using phenolphthalein as indicator.

A-3.2.2 Dilute the titrating solution obtained as in **A-3.2.1** to exactly 250 ml and mix. Take 50 ml of the solution, and add 5 ml of nitric acid, 50 ml of silver nitrate solution and 3 ml of nitrobenzene. Shake vigorously for 1 minute. Titrate the excess of silver nitrate solution with standard ammonium thiocyanate solution using ferric alum as indicator. Carry out a blank titration.

A-3.2.3 Take exactly 2 ml of the material in a dry separating funnel of 150 ml capacity. Add about 20 ml of chilled distilled water. Shake for 30 seconds and immediately separate the benzoyl chloride layer, extract the aqueous layer with 25 ml of solvent ether three times, separating ether after extraction. Titrate the aqueous layer with standard sodium hydroxide solution using phenolphthalein as indicator.

A-3.3 Calculations

A-3.3.1 Total acidity (benzoic acid and free hydrochloric acid calculated as benzoyl chloride), percent by mass (X) $= \frac{7{\cdot}028\ (\ 50\ N_1 - V_1 N_2\ )}{M}$

where

N_1 = normality of standard sodium hydroxide solution,

*Specification for rectified spirit (*revised*).

†Specification for nitric acid (*first revision*).

‡Specification for ether (*revised*).

V_1 = volume in ml of standard sulphuric acid used for the excess of standard sodium hydroxide solution (*see* **A-3.2.1**),

N_2 = normality of standard sulphuric acid, and

M = mass in g of the material taken for the test (*see* **A-3.2.1**).

A-3.3.2 Total chlorides (as benzoyl chloride), percent by mass (Y) $= \frac{70{\cdot}28\ (V_3 - V_2)\ N_3}{M}$

where

V_3 = volume in ml of standard ammonium thiocyanate solution used in the blank titration (*see* **A-3.2.2**),

V_2 = volume in ml of standard ammonium thiocyanate solution used for the excess of silver nitrate solution (*see* **A-3.2.2**),

N_3 = normality of standard ammonium thiocyanate solution, and

M = mass in g of the material taken for the test (*see* **A-3.2.1**).

A-3.3.3 Free chlorides (as benzoyl chloride), percent by mass (Z) $= \frac{14{\cdot}056\ V_4\ N_1}{V_5\ D}$

where

V_4 = volume in ml of standard sodium hydroxide solution used in the titration of aqueous layer (*see* **A-3.2.3**),

N_1 = normality of standard sodium hydroxide solution,

V_5 = volume in ml of the material taken for the test (*see* **A-3.2.3**), and

D = relative density of the material (*see* **A-2.3**).

A-3.3.4 Benzoyl chloride content, percent by mass $= Y - Z$

A-3.4 Determination of Free Hydrochloric Acid

A-3.4.1 Free hydrochloric acid, percent by mass $= 0{\cdot}260\ Z$

where Z = free benzoyl chloride content (*see* **A-3.3.3**).

A-3.5 Determination of Free Benzoic Acid

A-3.5.1 Free benzoic acid, percent by mass $= 0{\cdot}871\ (X - Y)$

where

X = total acidity (*see* **A-3.3.1**), and

Y = total benzoyl chloride content (*see* **A-3.3.2**).

A-4. DETERMINATION OF RESIDUE ON EVAPORATION

A-4.1 Apparatus

A-4.1.1 *Water-Bath*

A-4.1.2 *Silica Basin* — 7·5 cm diameter, about 2·0 cm deep.

A-4.1.3 *Oven* — capable of being maintained at 200 ± 5°C.

A-4.1.4 *Desiccator*

A-4.2 Procedure — Evaporate 10 ml of the material on the water-bath in the tared silica basin. Dry the residue for 2 hours in the oven at 200 ± 5°C. Cool the basin with the residue in the desiccator and weigh.

A-4.3 Calculation

$$\text{Residue on evaporation, percent by mass} = \frac{100\,(A - B)}{10\,D}$$

where

A = mass in g of the silica basin with the residue after evaporation and drying,

B = mass in g of the empty silica basin, and

D = relative density of the material (*see* **A-2.3**).

APPENDIX B

(*Clause* 6.1)

SAMPLING OF BENZOYL CHLORIDE, TECHNICAL

B-1. GENERAL REQUIREMENTS FOR SAMPLING

B-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination (*see also* **4.1**).

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost three-fourths filled by the sample.

B-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, batch number and other important particulars of the consignment.

B-2. SCALE OF SAMPLING

B-2.1 Lot — In a single consignment, all the containers of the same size containing material of the same grade drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, or of different grades, the containers belonging to the same batch and grade and of the same size shall be grouped together and each such group shall constitute a separate lot.

B-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FROM LOTS OF DIFFERENT SIZES

LOT SIZE	SAMPLE SIZE
N	n
(1)	(2)
Up to 15	3
16 „ 40	4
41 „ 65	5
66 „ 110	7
111 and above	10

NOTE — When the size of the lot is three or less, all the containers shall be sampled.

B-2.3 In order to ensure randomness of selection, the following procedure is recommended for use:

Arrange all the containers in the lot in a systematic manner and, starting from any one, count them as 1,2,....., up to r, and so on where r is the integral part of N/n (N and n being the lot size and sample size respectively). Every rth container thus counted shall be withdrawn to constitute the sample.

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 From each of the containers selected (*see* **B-2.2**) a representative portion of the material shall be drawn. From each of these individual portions, an equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample about 1 500 ml in volume. The composite sample shall be transferred to clean bottles and labelled with full identification particulars of the sample. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-4. TESTS

B-4.1 Tests for determination of the characteristics given in **3.1** and Table 1 shall be carried out on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For declaring the conformity of a lot to this specification, all the test results shall meet the corresponding specified requirements.

INDIAN STANDARDS

ON

Organic Chemicals (Miscellaneous) Materials

IS:

245-1970	Trichloroethylene, technical (*second revision*)
501-1963	Oxalic acid, technical and analytical reagent (*revised*)
716-1970	Pentachlorophenol (*first revision*)
717-1969	Carbon disulphide, technical (*first revision*)
718-1970	Carbon tetrachloride (*first revision*)
869-1969	Ethylene dichloride (*first revision*)
880-1956	Tartaric acid
3321-1965	Formaldehyde solution
4105-1967	Styrene (vinyl benzene)
4306-1967	Hexamethylenetetramine (hexamine)
4566-1968	Methylene chloride (dichloromethane), technical
5149-1969	Maleic anhydride, technical
5158-1969	Phthalic anhydride, technical
5254-1969	Acetanilide
5271-1969	Paraformaldehyde
5295-1969	Ethylene glycol
5296-1969	Chloroform, technical and analytical
5297-1969	Perchloroethylene (tetrachloroethylene), technical
5341-1969	Benzyl chloride, technical
5464-1970	Citric acid, monohydrate
5573-1969	Ethylene oxide
5591-1969	Chlorobenzene
5592-1969	Monochloroacetic acid
5992-1970	*p*-Dichlorobenzene, technical

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